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## DEVELOPMENT OF A LOW-VOC AIRCRAFT WHEEL WELL CLEANER

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### INTRODUCTION

Cleaning of wheel well areas and wing butts on US Navy aircraft is required to remove salt contamination as a corrosion control measure and remove buildup of soils to allow for proper inspection of the aircraft. Cleaners used for this purpose must conform to the performance requirements of military specification MIL-PRF-85570C Type V (Cleaning Compound, Aircraft, Exterior, Gel-Type Wheel Well Degreaser) which includes strict criteria for materials compatibility to protect common aircraft alloys and alkaline-sensitive polyimide-insulated wiring.

Cleaners previously used in navy aircraft wheel well cleaning suffered from several disadvantages environmentally. A primary concern is the aromatic solvent content, typically from 8.5 to 10 % by weight of the product. These compounds were cited in the 1990 Clean Air Act as Hazardous Air Pollutants (HAP) and have been targeted for elimination from aircraft maintenance operations. Aromatic solvents also have low Permissible Exposure Limits (PEL) and are not highly biodegradable.

E-class glycol ether coupling solvents, such as ethylene glycol monobutyl ether, are included in most products. Like aromatic solvents, these compounds are HAP and carry a low PEL (100 ppm). Other Volatile Organic Compounds (VOC) are used in these products which may have a total solvent content of up to 32 % by weight.

A final concern is the typical primary surfactant component in the aircraft cleaners, phenoxy ethoxylates. These surfactants contain a phenoxyl group in the chemical structure, which is thought to produce the toxic compound phenol as a product of degradation. This surfactant may result in a waste stream that can cause treatment problems or persistent waterway pollution if discharged to natural waterways. The revised specification does not directly preclude the use of these surfactants, but requires a minimum of 85% biodegradation by the EPA test for 28-day ultimate biodegradation.

At the start of this project, suppliers of MIL-C-85570B Type V were contacted and asked to submit products with less than 10% total solvent in the formulation for evaluation. Products with solvent levels below this level are considered aqueous cleaners according to California Air Resources Board (CARB) standards and as such are exempt from any VOC reporting requirements. Few responses were obtained and the performance of the products received was substandard. Therefore, a low-VOC control formulation was developed that could be either manufactured by the suppliers or used for comparison purposes for suppliers wishing to develop alternate formulations.

Potential solvents for inclusion in the formulation were selected based on environmental acceptability, safety considerations, and expected solvency as predicted by the reported Kauri-Butanol values. Solvents that performed well in initial evaluations were incorporated into a

water-rinsable gel formulation. The gel form is desired to allow the product to cling to vertical surfaces while the solvent dissolves the soil. The gel is ideally formed using surfactants, since the surfactants will aid in emulsification of the soil and make the final product water rinsable. The product must also not cause significant damage on aircraft structural materials.

This report details work sponsored by the Strategic Environmental Research and Development Program (SERDP) to develop environmentally preferred products for this application.

## EXPERIMENTAL

Accelerated Storage Stability. Approximately 50 ml of concentrated cleaning compound was poured into a clean 250 ml pressure resistant clear glass soda bottle approximately 24 cm in height and 6.5 cm in outside diameter. A strip of 1020 steel 6 x 0.5 x 0.02 in. was polished with 280-grit silicon carbide sandpaper, wiped with isopropyl alcohol, and placed in the bottle. The bottle was capped and inverted to completely coat the steel strip. The bottle containing the steel strip was placed in a hot water bath held at  $140 \pm 4^{\circ}\text{F}$  ( $60^{\circ}\text{C}$ ) for 8 hours and the bath allowed to cool for 16 hours. This cycle was repeated each day for a total of seven days. On the eighth day, the bottle was removed from the bath, uncapped, examined for separation, and the steel strip withdrawn and examined for corrosion. The aged sample was tested for cleaning efficacy and change in color by ASTM D1544, Color of Transparent Liquids (Gardner Color Scale).

Cleaning Efficacy, Method I. An aluminum panel coated with gloss white polyurethane topcoat conforming to MIL-PRF-85285C (Coating: Polyurethane, High-Solids) was sprayed with wire rope lubricant (Sprayon No. 201) or corrosion preventive compound conforming to MIL-C-81309 type II. The soil was wiped across the panel using a non-metallic aircraft cleaning and polishing pad to achieve uniform coverage. The soil was baked for 1 hour at  $220^{\circ}\text{F}$  ( $105^{\circ}\text{C}$ ). The panel was placed at a  $60^{\circ}$  angle from the horizontal and the wheel well cleaner applied to the panel using a pump spray applicator. After 5 minutes, the test panel was rinsed by spraying with distilled water using the same pump spray device. Ability to remove soil was determined qualitatively.

Cleaning Efficacy, Method II. Method II is identical to Method I with the exception that the soil was not baked, but allowed to dry at room temperature for 15 minutes. This method was selected for the C revision of MIL-PRF-85570.

Cloud Point. Cloud point was determined by placing approximately 100 ml of test solution in a 250 ml Erlenmeyer flask. An immersion thermometer was inserted through a rubber stopper so as to be suspended in the solution. The solution was heated and stirred continuously on a laboratory stir plate until clouding of the solution completely obscured the thermometer bulb. The flask was then placed on a cool stir plate with stirring continuing. The solution was allowed to cool and the cloud point was reported as the temperature that solution again became clear.

Consistency. The well of a consistometer (Central Scientific, Chicago, IL, Cat. No. 24925) was filled with 100 ml of wheel well cleaner. The gate was released and the extent of flow measured in centimeters was determined after 10 seconds.

Corrosion. Testing was performed in accordance with ASTM F483, Total Immersion Corrosion Test for Aircraft Maintenance Chemicals, ASTM F1110, Sandwich Corrosion Test, and ASTM F1111, Corrosion of Low-Embrittling Cadmium Plate by Aircraft Maintenance Chemicals.

Field-testing. Testing of the final product was performed on wheel wells and wing butts of in-service aircraft at Whidbey Island Naval Air Station, Washington.

Flash Point. Flash point was determined in accordance with ASTM-D93, Flash Point by Pensky-Martens Closed Cup Tester.

Foaming. 100 ml of solution was agitated for 2 minutes at 8000 rpm in a Warring blender. The solution was allowed to stand for 6 minutes and the remaining liquid was then measured.

Low Temperature Stability. 50 ml of concentrated cleaning compound was placed in a graduated cylinder and cooled to  $19 \pm 2$  °F ( $-8 \pm 1$  °C). The cylinder was then allowed to warm to room temperature, inverted 5 times, and examined for homogeneity.

Non-Volatile Content. Approximately 0.5 g of product was placed into each of three aluminum-weighing dishes of known weight and the sample weight recorded. The dishes were placed in a  $100 \pm 2$  °F ( $38 \pm 1$  °C) oven for 1 hour. The dishes were allowed to cool for 10 minutes in a desiccator and the final weight recorded.

pH. Sample pH was determined in accordance with ASTM E70, pH of Aqueous Solutions with the Glass Electrode.

Plastic Crazing. Testing was performed in accordance with ASTM F484, Stress Crazing of Acrylic Plastics in Contact with Liquid or Semi-Liquid Materials.

Polyimide Wire Degradation. Approximately 14 in. of wire conforming to MIL-W-81381B (Wire, Electric, Polyimide-insulated, Copper or Copper Alloy) was formed into a coil and placed in a 4 ounce (118 ml) wide mouth jar. The jar was filled with the cleaning compound, capped, and stored at room temperature for 14 days. Distilled water was run as a control. At the end of the storage period, the coil was removed and rinsed thoroughly with tap water. The coil was dried and wrapped tightly around a 0.125 inch (3 cm) mandrel and unwrapped slowly, noting the appearance and the number of cracks in the insulation.

Salt-Coated Surfaces, Effect on. Testing was performed according to ASTM F502, Effect of Cleaning and Chemical Maintenance Materials on Painted Aircraft Surfaces with the additional provision that panels were sprayed with ASTM D1141, Substitute Ocean Water, and dried in a  $100 \pm 4$  °F ( $38 \pm 2$  °C) convection oven prior to testing.

Unpainted Surfaces, Effect on. Testing was performed according to ASTM F485, Effects of Cleaners on Unpainted Aircraft Surfaces.

Water Content. Testing was performed according to ASTM D95, Water in Petroleum Products and Bituminous Materials by Distillation.

## RESULTS AND DISCUSSION

Accelerated storage stability. Prototype formulations caused corrosion of steel at the vapor-liquid interface in this test. Formulations containing 1-5% capryloamphopropionate (Monateric 1000), an amphoteric surfactant, were evaluated and found to be effective in inhibiting the corrosion at a concentration of approximately 5%.

Cleaning Efficacy. Preliminary evaluation of potential solvents on soiled panel is given in Table 4.1. Solvents were ranked visually from 1 (complete removal) to 4 (no effect) according to ability to remove wire rope lubricant and Mil-C-81309E (Corrosion Preventive Compounds, Water Displacing, Ultra-Thin Film).

**Table 4.1**  
**Relative Ranking of Solvent Performance by Method I**

<i>Solvent</i>	<i>Wire Rope Lubricant</i>	<i>Corrosion Preventive Compound</i>
D-limonene	1	1
Aromatic hydrocarbon, FP >150	1	1
Isoparaffin FP>200°F	2	1
Butyl Salicylate	1	2
Heptane	2	2
Ethylene glycol	3	4
Decanol	4	4
50% Neodol 91-6	4	4
MIL-C-85570B type V	1	1
MIL-PRF-85570C type V	2	2

Since D-limonene has desirable environmental characteristics and performed about as well as the original aromatic solvent base used in original formulation, it was chosen as the primary solvent base. Ternary phase diagrams of D-limonene, water, and surfactant, were prepared to determine regions of gel formation. Compositions containing 0-20 % D-limonene were studied. Stable gel formation was obtained in the regions of 0 to 20% D-limonene and 60 to 70% Neodol 91-8 (linear ethoxylate, HLB = 13.9) and 10-20% D-limonene and 55-65% Neodol 91-6 (HLB = 12.5). No regions of gel formation were found in the region of interest with Neodol 91-2.5 (HLB = 8.5) as the surfactant phase.

Complete soil removal could be obtained in the cleaning efficacy test with a gel product consisting of 10% D-limonene, 65% Neodol 91-8, and 25% water. This formulation had high temperature stability problems, but established a starting point for the product. Other components were then added to improve stability, flow properties, flash point, and corrosion.

Note that performance of MIL-PRF-85570C type V was inferior to the aromatic solvent-based gel of MIL-C-85570B type V. Although the performance of D-limonene was comparable to that of the aromatic solvent, the total solvent content of the new material was much lower overall, 10% compared to 32%, which affected the performance. The new D-limonene-based material did, however, completely remove the soils by cleaning efficacy method II in which the soils are dried at room temperature rather than baked.

Cloud point. Cloud points of initial prototype formulations were fairly low, which could cause separation problems if higher temperatures were experienced in storage. The breadth of the molecular weight distribution of the surfactants was increased and 1% by weight sodium bicarbonate added to improve high temperature stability of the gel.

Consistency. Prototype formulations that contained sufficient surfactant to properly emulsify the solvent into a stable gel were very stiff, making spraying much too difficult. The addition of dipropylene glycol sufficiently reduced the viscosity while maintaining the emulsion and gel characteristics. Moreover, the viscosity of the final control formulation could be reduced by the addition of a small quantity of dipropylene glycol or increased by the addition of Neodol 23-1.

Field-testing. Results of the field-testing were reported to be favorable. Although laboratory performance of the new material was not equivalent to the previous aromatic solvent-based material, the product performed well on operational soils encountered in actual use.

Flash Point. Due to storage and safety considerations, products should have a flash point of greater than 140°F (60°C). Above this threshold materials are classified as "combustible" rather than "flammable" and as such do not have special storage requirements. The flash point of the D-limonene, the solvent base of the material, is 119°F (48 °C). When incorporated into the formulation at even less than 10%, the flash point of the formulated product was 125°F (91°C). This problem was corrected by replacing part of the solvent phase with a high flash (>200°F [>93°C]) isoparaffin hydrocarbon, Isopar M. Reducing the D-limonene content to 7% and adding 3% Isopar M, increased the flash point of the formulation to 140°F (60°C).

Sandwich Corrosion. Prototype formulations had an effect on aluminum during the sandwich corrosion test. Fluoboric acid, mercaptobenzothiazole, sodium molybdate, and benzotriazole were evaluated as potential inhibitors. Testing showed that 0.5% benzotriazole was effective in minimizing the effect on aluminum.

Final formulation. The final version of the control formulation for MIL-PRF-85570 Type V is given in Table 4.2. Parts A and B are prepared separately, then part B is slowly added into part A. Complete test results with this formulation are given in Table 4.3.

**Table 4.2**  
**MIL-PRF-85570 Type V Control Formulation**

<i>Component</i>	<i>Parts by weight</i>	<i>Supplier</i>
Part A:		
Neodol 23-1	5.0	Shell
Neodol 91-2.5	5.7	Shell
Neodol 91-6	10.6	Shell
Neodol 91-8	10.7	Shell
Monateric 1000	4.9	Mona
Prep-solve <sup>1</sup>	6.7	Glidco
Isopar M	2.9	Exxon
Dipropylene Glycol	9.0	Fisher
Benzotriazole	0.5	Fisher
Part B:		
Sodium Bicarbonate	1.0	Fisher
Deionized water	43.0	N/A
Total	100.0	

<sup>1</sup>D-limonene with proprietary anti-oxident



**Table 4.3**  
**MIL-PRF-85570 Type V Control Formulation Test Results**

<i>PROPERTY</i>	<i>REQUIREMENT</i>	<i>RESULT</i>
Accelerated storage stability	No separation or effect on steel strip	No separation or effect on steel strip
Appearance	Homogenous, no foreign matter	Homogeneous, no foreign matter
Biodegradability	85.0 % in 28 days, min	87.0 % in 28 days
Cadmium corrosion (mg/cm <sup>2</sup> /day)	0.20 mg/cm <sup>2</sup> /day, max	0.008 mg/cm <sup>2</sup> /day
Cleaning efficacy	100% removal of wire rope lube	100% removal of wire rope lube
Cloud point	120°F, min	145°F (melting point = 106°F)
Consistency	13 to 23 cm flow in 10 seconds	17 cm flow in 10 seconds
Flash point, closed cup	140°F, min.	140°F
Hydrogen Embrittlement	No breakage at 150 hrs.	No breakage at 150 hrs.
Low temperature stability	No separation	No separation
Non-volatile content	Informational	47.37 %
pH	7.0 – 10.0	8.95
Plastics, effect on Acrylic, type A Acrylic, type C Polycarbonate, MIL-P-83310	No crazing after 4 hours No crazing after 8 hours No crazing after 2 hours at 2000 psi	No crazing after 4 hours No crazing after 8 hours No crazing after 2 hours at 2000 psi
Polyimide insulated wire, effect on	No dielectric leakage, no effect greater than distilled water	No dielectric leakage, no effect greater than distilled water
Salt-coated surfaces, effect on	No non-rinseable film	No non-rinseable film
Sandwich corrosion, aluminum 2024 bare 2024 clad 7075 bare 7075 clad	Rating 1 max., or no effect greater than DI water control	Rating (DI water control rating) 1 (1) 1 (1) 2 (2) 1 (1)
Solvent content	<10%	6.96 ± 0.57 %
Sprayability	Uniform spray, clings to vertical panel	Uniform spray, clings to vertical panel
Storage stability	No separation or effect on steel strip	No separation or effect on steel strip
Total immersion corrosion Al Ti Steel Mg	max loss, mg/cm <sup>2</sup> /day 0.04 0.04 0.04 0.20	mg/cm <sup>2</sup> /day 0.001 0.001 0.003 0.094
Unpainted surfaces, effect on	No streaking or staining	No streaking or staining
Water content	Informational	45.67 %

## CONCLUSIONS

1. An aircraft wheel well cleaning gel has been developed based on D-limonene, isoparaffinic hydrocarbon, and surfactants, which meets the requirements of Mil-PRF-85570C Type V. Laboratory and field-testing suggest that this product has good performance characteristics and will be a suitable replacement for aromatic solvent-based products previously used in this application.